

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2001-209925

(43)Date of publication of application : 03.08.2001

(51)Int.Cl.

G11B 5/73
C23C 18/32
C23C 28/00
C25D 11/04
C25D 11/18
G11B 5/84

(21)Application number : 2000-017073

(71)Applicant : NIPPON LIGHT METAL CO LTD

(22)Date of filing : 26.01.2000

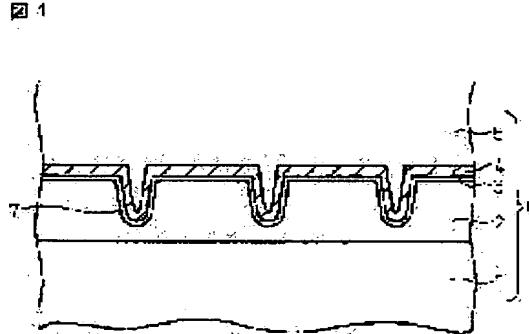
(72)Inventor : KOBAYASHI YASUSHI
TAKEBAYASHI YASUSHI
SHIMADA HIDEKI
TAKAHASHI TAKEYOSHI

(54) ALUMINUM SUBSTRATE FOR MAGNETIC RECORDING MEDIUM AND METHOD FOR PRODUCING SAME

(57)Abstract:

PROBLEM TO BE SOLVED: To provide an aluminum substrate for a magnetic recording medium having high impact resistance and high adhesion of a P-containing Ni alloy plating film to the substrate.

SOLUTION: The aluminum substrate has a P-containing Ni alloy plating film formed by electroless plating on the surface of an aluminum base. In detail, the aluminum substrate has an anodic oxide coating on the surface of the aluminum base, a catalytic nuclear metal deposited on the surface of the coating, an under metallic plating film formed by flash plating, substantially covering the coating with the deposited catalytic nuclear metal and having a composition different from that of the catalytic nuclear metal and that of the P-containing Ni alloy plating film and the P-containing Ni alloy plating film formed by electroless plating on the under metallic plating film.



LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

- [Date of registration]
- [Number of appeal against examiner's decision of rejection]
- [Date of requesting appeal against examiner's decision of rejection]
- [Date of extinction of right]

Copyright (C); 1998,2003 Japan Patent Office

*** NOTICES ***

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.**** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

CLAIMS

[Claim(s)]

[Claim 1] In the aluminum substrate which equipped with the electroless deposition film of a P content nickel alloy the base material side which consists of aluminum The base material which consists of aluminum, and the porous anodic oxide film generated by anodic oxidation of this aluminum base material, The substrate metal flash plate plating film of a presentation which is different from said P content nickel alloy electroless deposition film in said porous anodic oxide film with which the metal used as the catalyst nucleus which deposited in this porous anodic oxide film side, and the metal used as this catalyst nucleus deposited unlike the presentation of the metal which serves as the wrap aforementioned catalyst nucleus substantially, The aluminum substrate for magnetic-recording media characterized by having said P content nickel alloy film which carried out electroless deposition to this substrate metal flash plate plating film.

[Claim 2] The aluminum substrate for magnetic-recording media according to claim 1 with which thickness of the porous anodic oxide film generated by said anodic oxidation is characterized by being 0.5–50 micrometers.

[Claim 3] The aluminum substrate for magnetic-recording media according to claim 1 or 2 characterized by said porous anodic oxide film being an anodic oxide film by oxalic acid or the chromic-acid electrolytic solution.

[Claim 4] The aluminum substrate for magnetic-recording media given in any 1 term to claims 1–3 characterized by being the alloy which uses as a principal component 1 or two or more metals with which the metal used as the catalyst nucleus which deposited in said porous anodic oxide film side was chosen from Pd, Cu, Ag, Sn, P, nickel, or these metals.

[Claim 5] Moreover, for P content, the thickness is [said substrate metal flash plate plating film] an aluminum substrate for magnetic-recording media given in any 1 term to claims 2–4 to which it is characterized by being a P content nickel alloy below more than 3wt%10wt% at 5nm or more.

[Claim 6] Moreover, for P content, the thickness is [said P content nickel alloy electroless deposition film] the aluminum substrate for magnetic-recording media according to claim 5 to which it is characterized by being 10 – 13wt% at 0.5 micrometers or more.

[Claim 7] The base material which consists of aluminum, and the porous anodic oxide film whose thickness generated by anodic oxidation by the oxalic acid or the chromic-acid electrolytic solution of this aluminum base material is 0.5–50 micrometers, The P content nickel alloy substrate metal flash plate plating film whose P content wrap thickness is less than [more than 3wt%10wt%] in 5nm or more substantially about said porous anodic oxide film with which Pd and Pd which deposited in said porous anodic oxide film side deposited, The aluminum substrate for magnetic-recording media characterized by equipping P content with the P content nickel alloy film whose wrap thickness is 10 – 13wt% in 0.5 micrometers or more about this metal membrane.

[Claim 8] In the manufacture approach of the aluminum substrate equipped with the electroless deposition film of a P content nickel alloy The process which performs anodizing to the base material which consists of aluminum, and forms a porous anodic oxide film, The process which forms in this porous anodic oxide film side the substrate metal flash plate plating film of a presentation which is different from said P content nickel alloy electroless deposition film in said

porous anodic oxide film with which the process which deposits the metal used as a catalyst nucleus, and the metal used as this catalyst nucleus deposited unlike the presentation of the metal which serves as the wrap aforementioned catalyst nucleus substantially, The manufacture approach of the aluminum substrate for magnetic-recording media characterized by passing through the process which forms said P content nickel alloy electroless deposition film in said porous anodic oxide film in which this substrate metal flash plate plating film was formed, and each process which grinds this P content nickel alloy electroless deposition film.

[Claim 9] The manufacture approach of the aluminum substrate for magnetic-recording media according to claim 8 characterized by performing said anodizing in oxalic acid or the chromic-acid electrolytic solution.

[Claim 10] The manufacture approach of the aluminum substrate for magnetic-recording media according to claim 8 or 9 characterized by being the alloy which uses as a principal component 1 or two or more metals with which the metal used as the catalyst nucleus which deposited in said porous anodic oxide film side was chosen from Pd, Cu, Ag, Sn, P, nickel, or these metals.

[Claim 11] The process which performs anodizing to the base material which consists of aluminum in oxalic acid or the chromic-acid electrolytic solution, The process which deposits Pd in the porous anodic oxide film side generated by this anodic oxidation, The process from which thickness forms the P content nickel alloy substrate metal flash plate plating film whose phosphorus content is less than [more than 3wt%10wt%] in said porous anodic oxide film with which this Pd deposited by 5nm or more, The process in which a phosphorus content forms the P content nickel alloy electroless deposition film whose thickness is 10 – 13wt% in 0.5 micrometers or more about said porous anodic oxide film with which this substrate metal flash plate plating film was formed, The manufacture approach of the aluminum substrate for magnetic-recording media characterized by passing through each process which grinds this P content nickel alloy electroless deposition film.

[Translation done.]

*** NOTICES ***

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.**** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]**[0001]**

[Field of the Invention] This invention relates to the aluminum substrate for magnetic recording media equipped with the P content amorphous nickel alloy film on the aluminum (aluminum contains pure aluminium and aluminium alloy unless it shows clearly especially in this specification .) base side for high recording density magnetic recording media , such as a cover half thin film magnetic recording disk (hard disk) use for electronic information processing , and the manufacture approach of the substrate .

[0002]

[Description of the Prior Art] The fixed magnetic recording medium (magnetic disk drive) called a "hard disk drive" or a "hard disk" as the external storage is used for information processors, such as a computer. Generally, the aluminum substrate is used as a substrate of the record medium (magnetic disk) of this fixed magnetic recording medium. After this magnetic-recording medium graduates a nonmagnetic aluminum base material by grinding, usually performs radio solution nickel-P alloy plating after surface defecation and zinc permutation processing, and it graduates it by polish further and it performs texture ring processing, it carries out sequential formation of a non-magnetic metal substrate layer membrane, a magnetic film, a protective coat, and the lubricating film, and is manufactured.

[0003] Since it corresponds to the hard disk equipment of a portable type carried in a luggable computer etc. in recent years, the shock resistance required of a magnetic disk is also becoming high every year so that it may not get damaged by the collision with the magnetic head at the time of actuation and migration. In connection with it, shock resistance is becoming inadequate with the substrate which prepared the P content nickel alloy plating film in the aluminum base material as mentioned above. Then, instead of an aluminum substrate, it is expensive, and although workability is inferior, the glass substrate excellent in shock resistance is beginning to be used.

[0004] In many cases, along with the substrate circumferential direction, the concentric circular texture ring is given to the aluminum substrate which prepared the no electrolyzing nickel or non-electrolyzed P content nickel alloy plating film by polish on the front face. This is for making a magnetic anisotropy increase while preventing adsorption between the magnetic-recording head for record playback, and a magnetic-recording medium. Furthermore, recently, forming a minute projection in the CSS zone of a substrate by laser beam machining is also performed with remarkable reduction of the head flying height at the time of actuation.

[0005] However, in the case of a glass substrate, since it is hard and workability is bad, it is difficult [it] to give a texture ring. And since [that the absorptivity of a laser beam is bad and] it is generally transparent, and melting temperature is also remarkable and high, it is also difficult to form much projections of a specific configuration in homogeneity. As a shock-proof high substrate which a texture ring is possible and can form a minute projection in homogeneity by laser beam machining as the cure, Pd is made to stick to a glass side, and although the glass substrate which forms the P content nickel alloy plating film on it is proposed (surface technical Vol 44 831-835 page 1993), workability is inferior to productivity in glass bad.

[0006] An aluminum substrate is cheap, and workability is good, and shock resistance is inadequate although productivity is good. The good aluminum substrate of adhesion is proposed by the substrate of the P content nickel alloy plating film which was filled up with the metal, considered as the catalyst nucleus, and formed the P content nickel alloy plating film in strong acidity liquid on it into the micropore of the anodic oxide film of aluminum or aluminum alloy at JP,62-3423,A.

[0007]

[Problem(s) to be Solved by the Invention] When the aluminum substrate proposed by said JP,62-3423,A is examined, the processing which forms the P content nickel alloy plating film for the anodic oxide film of the aluminum filled up with the metal in strong acid liquid into micropore dissolves an anodic oxide film remarkably, and it produces unevenness in the thickness of film, and it not only reduces the smooth nature on the front face of a coat, but when extreme, a coat will be dissolved and removed completely partially. For this reason, the external waviness property on the front face of a substrate after polish does not need to be acquired, or for obtaining the smooth P content nickel alloy plating film, it is necessary to enlarge the amount of polishes of the plating film.

[0008] Moreover, since the plating liquid containing P beyond 10wt% used as substrate plating film for magnetic disks for nickel-P alloys has the high reactivity in early stages of a deposit, adhesion with the good case where an anodic oxide film is deposited directly is not acquired.

[0009]

[Means for Solving the Problem] Namely, the 1st invention is set to the aluminum substrate which equipped with the electroless deposition film of a P content nickel alloy the base material side which consists of aluminum. The base material which consists of aluminum, and the porous anodic oxide film generated by anodic oxidation of this aluminum base material, The substrate metal flash plate plating film of a presentation which is different from said P content nickel alloy electroless deposition film in said porous anodic oxide film with which the metal used as the catalyst nucleus which deposited in this porous anodic oxide film side, and the metal used as this catalyst nucleus deposited unlike the presentation of the metal which serves as the wrap aforementioned catalyst nucleus substantially, It is the aluminum substrate for magnetic-recording media characterized by having said P content nickel alloy film which carried out electroless deposition to this substrate metal flash plate plating film. By considering as such structure, an aluminum substrate with high shock resistance and the high adhesion over the substrate of the P content nickel alloy plating film is obtained.

[0010] The porous anodic oxide film generated by anodic oxidation of an aluminum base material is low rather in respect of a degree of hardness in whether it is almost comparable as the P content amorphous nickel alloy film. However, since it is hard to carry out elastic deformation of the elastic modulus very highly, and there is almost no elongation, and yield stress is also large and it is hard to produce plastic deformation, a bigger load can be borne. A shock-proof high aluminum substrate is obtained by setting thickness of this porous anodic oxide film to 0.5 micrometers or more.

[0011] Furthermore, when this anodic oxide film is a porous anodic oxide film by oxalic acid or the chromic-acid electrolytic solution, in addition to the above-mentioned property, the aluminum substrate which has thermal resistance without bulging in a magnetic-recording disk manufacture process is obtained. Furthermore, an aluminum substrate with the higher adhesion over the substrate of the P content nickel alloy plating film is obtained by considering as the alloy which uses as a principal component 1 or two or more metals which were chosen in the metal which deposits this porous anodic oxide film side from Pd, Cu, Ag, Sn, P, nickel, or these metals.

[0012] Furthermore, when the thickness moreover uses the substrate metal flash plate plating film as nickel alloy plating film by 5nm or more, an aluminum substrate with the higher adhesion over a substrate is obtained. Furthermore, when P content moreover makes [the thickness] the P content nickel alloy electroless deposition film 10 – 13wt% by 0.5 micrometers or more, a nonmagnetic aluminum substrate with a high degree of hardness is obtained.

[0013] In the manufacture approach of an aluminum substrate that the 2nd invention was

equipped with the electroless deposition film of a P content nickel alloy. The process which performs anodizing to the base material which consists of aluminum, and forms a porous anodic oxide film. The process which forms in this porous anodic oxide film side the substrate metal flash plate plating film of a presentation which is different from said P content nickel alloy electroless deposition film in said porous anodic oxide film with which the process which deposits the metal used as a catalyst nucleus, and the metal used as a catalyst nucleus deposited unlike the presentation of the metal used as said catalyst nucleus. It is the manufacture approach of the aluminum substrate for magnetic-recording media characterized by passing through the process which forms said P content nickel alloy electroless deposition film in said porous anodic oxide film in which this substrate metal flash plate plating film was formed, and each process which grinds this P content nickel alloy film. By considering as such a manufacture approach, a porous anodic oxide film side is hard to dissolve, the amount of polishes of the P content nickel alloy electroless deposition film can be lessened, and an aluminum substrate with shock resistance productivity is good and high and the high adhesion over the substrate of the P content nickel alloy electroless deposition film can be manufactured.

[0014] By setting thickness of this porous anodic oxide film to 20 micrometers or more, a shock-proof high aluminum substrate can be manufactured further. In addition to the above-mentioned effectiveness, by performing this anodizing in oxalic acid or the chromic-acid electrolytic solution, an aluminum substrate without bulging can be manufactured in a magnetic-recording disk manufacture process.

[0015] In addition to the above-mentioned effectiveness, an aluminum substrate with the higher adhesion over the substrate of the P content nickel alloy plating film can be manufactured by depositing the alloy which uses as a principal component 1 or two or more metals which were chosen as this porous anodic oxide film side from Pd, Cu, Sn, Ag, nickel, or these metals. When it is weak alkali - the acescence preferably and thickness sets to 5nm or more, in addition to the above-mentioned effectiveness, a porous anodic oxide film side dissolves the plating liquid of the process which forms this substrate metal flash plate plating film -- having -- hard -- the amount of polishes of the P content nickel alloy electroless deposition film -- few -- it can do -- production -- good -- shock resistance -- an aluminum substrate with the adhesion it is high and high over the substrate of the P content nickel alloy electroless deposition film can be manufactured.

[0016] The plating liquid of the process which forms this P content nickel alloy electroless deposition film is acidity, in addition to the above-mentioned effectiveness, a porous anodic oxide film side is hard to dissolve, and thickness can manufacture an aluminum substrate with a high degree of hardness, when [amorphous] a phosphorus content is 10 - 13wt% in 0.5 micrometers or more.

[0017]

[Embodiment of the Invention] The aluminum substrate 6 which drawing 1 shows 1 operation gestalt of the aluminum substrate for the magnetic-recording media of this invention, and is shown here. This aluminum is anodized on the aluminum base material 1. Sequential formation of the wrap P content nickel alloy electroless deposition film 5 is carried out [porous anodic oxide film / 2 / with which the formed porous anodic oxide film 2, the metal 3 used as the catalyst nucleus deposited on this coat 2, and the metal 3 used as this catalyst nucleus deposited] in the wrap substrate metal flash plating film 4 and this substrate metal flash plating film 4. The substrate metal plating film 4 is a metal with which the P content nickel alloy plating film 5 differs from a presentation unlike the metal 3 used as a catalyst nucleus here. 7 is a hole in an anodic oxide film 2 (pore).

[0018] although the presentation is not limited as an aluminum base material -- the other general aluminum-2-5wt%Mg system alloy of aluminum -- a solid-solution-strengthening type -- it is -- a crystallization object -- little mechanical grant on the strength can be performed. The porosity anodic oxide film 2 is formed by the direct current anodizing process by making an aluminum base material into an anode plate in acid liquid. Shock resistance is high hard, and these anodic oxidation coatings 2 cover the elasticity nature of an aluminum base material, and case [like a portability magnetic-recording medium], they become desirable. If especially this

thickness is set to 0.5 micrometers or more, elasticity ** of an aluminum base material will be covered and shock resistance will be given. It is desirable to exceed 20 micrometers preferably. [0019] Although a magnetic-recording disk forms an aluminum substrate for Cr film after texture processing by the spatter and forms magnetic-recording media, such as Co, in the law further, since an aluminum substrate carries out the temperature up of it to 250–300 degrees C in this case, there are few the water of crystallization and the amounts of attached groundwater in this coat that this porous anodic oxide film is oxalic acid or a chromic-acid anodic oxide film, and it can be equal to said heating, can give thermal resistance, and is desirable.

[0020] Although especially the surface roughness of an anodic oxide film 2 is not limited, since will plate thickly the thickness of the P content nickel alloy electroless deposition film, an aluminum substrate side will be ground, polish takes time amount and cost and productivity worsen when too large, when surface roughness considers as 500A or less by Ra, it is desirable. Combine with the substrate metal flash plating film 4 plated on it firmly, and the catalyst nucleus metal 3 deposited on the porosity anodic oxide film 2 stops being able to exfoliate easily, and is excellent in endurance. Especially this catalyst nucleus metal 3 deposits in homogeneity in comparison, and is firmly combined with it being the alloy which uses as a principal component 1 chosen from Pd, Ag, Sn, P, Cu, nickel, or these, or two sorts or more with the substrate metal flash plating film 4 at the 2nd page of a porosity anodic oxide film. here -- a principal component -- these metals -- more than 50wt% -- it says containing. this becoming firm -- the front face of a porosity anodic oxide film, and a hole -- it is thought that it is based on the anchor effect by the substrate metal flash plate plating film depositing inside. it is especially Pd -- catalyst nature -- high -- a hole -- an inner deposit is also fully performed and is desirable.

[0021] By the thickness of the substrate metal flash plating film 4 being nickel alloy 5nm or more, and making P content into less than [more than 3wt%10wt%], for giving sufficient peeling resistance, giving toughness, and the thickness's being 0.5 micrometers or more about the P content nickel alloy electroless deposition film 5, and making P content into 10 – 13wt%, it will join together firmly, will have a degree of hardness, and will have nonmagnetic. And by being referred to as 0.5 micrometers or more, texture processing of the film thickness of the P content nickel alloy electroless deposition film 5 can be carried out.

[0022] the aluminum substrate considered as such a configuration -- adhesion -- peeling resistance and shock resistance are highly excellent. After this, an aluminum substrate carries out texture processing and prepares Cr, Ti, nickel, Si, Ta, W, or the film used as the alloy which uses at least one sort as a principal component in these as nonmagnetic substrate film.

[0023] On this nonmagnetic substrate film, a magnetic film is prepared for the alloy which uses Co or Co as a principal component by approaches, such as a spatter. On this magnetic film, film, such as carbon, is prepared as a protective coat, and it considers as the substrate for the magnetic-recording playback made from aluminum. Next, the manufacture approach is explained.

[0024] In order to pierce aluminum plates, such as an aluminum-Mg system containing pure aluminum or 2–5wt%Mg, with a press, to create a doughnut-like aluminum plate and to remove a press peculiarity, laminating pressurization is carried out and the pierced doughnut-like aluminum plate is heated at 250–350 degrees C. Grinding is carried out in order to take out flat since then. one sort or two sorts or more of acids chosen from the group which consists of inorganic [of a sulfuric acid, oxalic acid, phosphoric acid, a chromic acid, and a tartaric acid] and an organic acid after pretreatment of cleaning, acid cleaning, backwashing by water, etc. after grinding are included -- if direct-current anodizing is preferably performed by making an aluminum substrate into an anode plate in a with a pH of three or less strong acid nature water solution, a porosity mold anodic oxide film will be obtained. Although the concentration of an acid changes with ****, it is 0.5 – 35% in general. If temperature is preferably made into low temperature at 5–30 degrees C, its coat degree of hardness will increase. The thickness of film is controllable by seal-of-approval quantity of electricity. There is no effect to the shock resistance of a coat with big class of acid and electrolytic condition. Although there is no need after anodizing, the so-called thickness of the nature oxide film of nonporous in the low section of an anodizing coat and a barrier layer may be equalized. Therefore, the approach of lowering the seal-of-approval electrical potential difference after anodizing termination in the same electrolytic solution of

anodizing, for example etc. may be given. Or among an after [anodizing] neutral solution, in the neutral salt electrolytic solution of pH 4–9, a pressure up may be carried out to 50V by electrolysis with constant current, and the nature anodic oxidation of nonporous whose thickness is about 70nm may be desirably established between an aluminum base material and a porosity anodic oxide film. moreover, the hole by secondary electrolytic deposition -- metal deposit processing inside may be performed.

[0025] The processing by oxalic acid or chromic-acid electrolysis can make low water content in the anodic oxide film obtained, and is desirable. Moreover, the obtained coat has thermal resistance and the aluminum substrate which has thermal resistance without bulging in a magnetic-recording disk manufacture process is obtained. The metal used as the catalyst nucleus after anodizing is deposited. Before depositing the metal used as a catalyst nucleus, alkali, such as acids, such as a nitric acid and fluoric acid, or caustic alkali of sodium, may wash this anodizing base material. Mechanical polish may be carried out with the Taira grinding stone, a brush, shot blasting, a buff, etc., and you may etch and grind from acids, such as a nitric acid and fluoric acid, surface roughening is etched and carried out by the chemical approach, surface roughening is carried out by the mechanical approach, or law is both adopted, and surface roughness is adjusted. make it any -- in Ra, preferably, still more preferably, if the average of roughness height of a porosity anodic oxide film side considers as 50A or less, it may raise adhesion with the P content nickel alloy electroless deposition film, and lessens the 100A or less of the 500A or less of the amounts of polishes. When grinding polish is carried out, removal washing of the grinding polish slag is carried out after that. 100 degrees C – 300 degrees C heat treatment may be performed for distortion picking, dehydration, degasifying, or crystallization between the finishing polishes after [after aluminum base material grinding to] P content nickel alloy electroless deposition processing.

[0026] A deposit of the catalyst nucleus metal to an anodic oxide film can adopt an electrolytic decomposition process or the catalyst giving method to a nonconductor top. For example, it is immersed into the water solution of a catalyst nucleus metal salt, and the aluminum base material which carried out anodizing is deposited in a porous anodic oxide film side by metal electrolysis, no electrolyzing, or adsorption. In this case, the aluminum base material which carried out anodizing is immersed into the water solution of the metal which has a reduction operation as pretreatment of a deposit of a catalyst nucleus metal, and the reducibility metal is made to once adhere to a porous anodic oxide film side. It is immersed into the water solution of said metal salt since then by this reducing power, and a catalyst nucleus metal is returned in respect of a porous anodic oxide film. Consider as a catalyst nucleus metal or Or an aluminum base material is immersed into the water solution of a catalyst nucleus metal salt, a metal salt is made to adhere to a porous anodic oxide film side, without performing the aforementioned pretreatment, and the approach of using as a catalyst nucleus metal with heating or other means etc. can be adopted.

[0027] Especially although especially the metal used as the catalyst nucleus to deposit is not limited, it is the alloy which uses as a principal component 1 chosen from Pd, Cu, Ag, Sn, P, nickel, or these, or two sorts or more, and like ****, the aluminum base material which carried out anodizing after said grinding washing into water solutions, such as these chlorides and a nitrate, is immersed, and this catalyst nucleus metal is deposited in a porous anodic oxide film side.

[0028] After washing the aluminum base material with which the catalyst nucleus metal deposited, formation of the flash plating film to plate In the alkali dissolved in high concentration, or an acid water solution, comparatively a metal salt for a short time by for example, the formation approach plated with the time amount for about 5 – 180 seconds By covering the whole porous anodic oxide film surface where the catalyst nucleus metal deposited the metal of a presentation which is different from the surface P content nickel alloy electroless deposition film by such approach unlike the presentation of the metal used as a catalyst nucleus Without dissolving a porous anodic oxide film with the electroless deposition processing liquid of degree process, plating liquid cannot be soiled with the deposited catalyst nucleus metal, and electroless deposition processing can be carried out in the pure condition. The plating liquid of flash plating

can process without polluting P content nickel alloy electroless deposition liquid as it is the plating liquid containing nickel salt and P salt. If it plates with whenever [low-temperature], for example, 20–50 degrees C, comparatively, whenever [solution temperature / of flash plating] can suppress the dissolution of substrate material, and can lower contamination of plating liquid. When acidity or alkalinity of this flash plating liquid is set to 4.0–10.0 with weak alkali thru/or weak acidic liquid, for example, pH display, there are few amounts of dissolutions of a porosity mold anodic oxide film, and a coat loses smoothing and the oxide film itself and is desirable. When carrying out flash plating of the P content nickel alloy, as a plating bath, the thing containing sources of nickel, such as a nickel sulfate and a nickel chloride, and *****, such as hypophosphite, can be adopted, for example.

[0029] The base material in which the flash plating film was formed forms the P content nickel alloy film by the electroless deposition method next. The amorphous nickel alloy film of P content is obtained by this electroless deposition. Here, with the P content amorphous nickel alloy film by which electroless deposition was carried out, what contained the 3rd element, for example, Cu, in addition to nickel and P is included. In order to form this P content amorphous nickel alloy film, acidity or alkalinity is made into strong acid nature, **** concentration is raised, and the P content amorphous nickel alloy film is formed. The source of nickel and the source of P can adopt a thing including sources of nickel, such as a nickel sulfate and a nickel chloride, and sources of P, such as hypophosphite, as plating liquid, for example. The source of nickel chooses concentration and 4 g/L – 8 g/L and the source of P choose 20 g/L – 40 g/L and temperature from 60 degrees C – 100 degrees C suitably as hypophosphorous acid.

[0030] For setting thickness of the P content amorphous nickel alloy film to 0.5 micrometers or more, the processing time can be made into 3 minutes or more, and it can carry out by managing a plating liquid presentation and a deposit rate making P content 10 – 13wt%.

[0031]

[Example] (Example 1) After using and carrying out distorted annealing of the aluminum base material of the shape of a doughnut which pierced the aluminum–4.5wt%Mg alloy plate with the press machine and sending display flatness, it degreased by lye, and the surface oxide was removed, rinsed and defecated with sulfuric-acid acidity liquid. Subsequently, anodizing was carried out on the following conditions and the porous anodic oxide film was formed.

Homogenization of the barrier layer after coat formation was carried out.

<Anodizing condition> electrolytic-solution presentation 5wt% oxalic acid water-solution direct-current electrolysis with constant current 1.2 A/dm² thickness of film Whenever [20 micrometer solution temperature] The catalyst nucleus metal was deposited on the following conditions after 3 times rinsing with pure water by the 30 degree-C setting following **.

<Catalyst nucleus metal deposit condition> liquid presentation PdCl₂ 5 g/LHCl Whenever [10 mL/L solution temperature] 20 degree-C setup time Subsequently ultrasonic rinsing processing was carried out for 30 seconds, and flash plating was carried out on the following conditions after defecation.

<Flash plating processing condition> liquid presentation NiSO₄ and 6H₂O 25 g/L hypophosphorous acid 25 g/LpH Whenever [7 solution-temperature] 40 degree-C time amount Subsequently it rinsed for 15 seconds and electroless deposition was carried out on the following conditions after defecation.

<Radio solution processing condition> liquid presentation NiSO₄ and 6H₂O 26g / L hypophosphorous acid 30 g/LpH Whenever [4.5 solution-temperature] 90 degree-C time amount Subsequently buffing was carried out for 120 minutes, 3 micrometers was removed, and surface roughness was finished.

[0032] Thus, the variation in adhesion, shock resistance, display flatness, and the thickness of anodic oxidation coatings was measured by the following approach about the produced aluminum substrate. Moreover, it heated for 10 seconds at 270 degrees C, and blistered by visual observation, and the existence of generating was inspected. A result is shown in Table 1.

<adhesion> JIS K Slitting was put in until it arrived at the aluminum ground with the cutter knife so that it might be set to several 9 of the 5400 (1990) coating commercial-test approach clearance spacing of 5mm, and a measure eye, the tape was stuck on it, this tape was removed

rapidly, and the condition that the front face separated was expressed as the evaluation mark 0–10 of a cross cut adhesion test (it expresses that adhesion is so good that evaluation mark are high).

<Shock-proof> Yoshida Seiki, Inc. make An impact was given to the substrate side using the pendulum type impact tester (form PST- 300), and the visual judgment of the existence of a depression was carried out.

<Display flatness> MIZOJIRI OPTICAL Co., Ltd. make Display flatness was measured every horizontal of a substrate using the interference fringe display flatness tester (form FT- 100).

<Variation of the thickness of anodic oxidation coatings> JIS H Product made from 8680-2 (1998) anodic-oxidation coating thickness determination approach Fischer INSU Vine Face It measured about the thickness of anodic oxidation coatings using the thickness measurement machine (form Fischer scope MMS).

[0033] A result is shown in Table 1.

(Example 2) Anodizing was carried out to the aluminum base material of the shape of a doughnut of this presentation on the same conditions as an example 1, and the porous anodic oxide film was formed. Homogenization of the barrier layer after coat formation was carried out.

Subsequently, the catalyst nucleus metal was deposited on the following conditions after rinsing by these conditions.

<Catalyst nucleus metal deposit conditions> (susceptibility-ized processing)

Liquid presentation SnCl₂ 10 g/LHCl Whenever [10 mL/L solution temperature] 20 degree-C setup time 30 seconds (activation)

Liquid presentation PdCl₂ 5 g/LHCl Whenever [10 mL/L solution temperature] 20 degree-C setup time Subsequently ultrasonic rinsing processing was carried out for 30 seconds, and flash plating was carried out on the same conditions as the example 1 after defecation.

[0034] Subsequently, it rinsed and electroless deposition was carried out on the following conditions after defecation.

<Electroless deposition processing condition> liquid presentation nickel (sulfuric acid nickel) 3.0 g/L hypophosphorous acid 30 g/LCu (sulfuric acid Cu) 0.6 g/LpH Whenever [8.8 solution-temperature] 90 degree-C time amount Subsequently buffing was carried out for 120 minutes, 3 micrometers was removed, and surface roughness was finished.

[0035] Thus, the property same about the produced aluminum substrate as an example 1 was measured by the same approach. A result is shown in Table 1.

(Example 3) Anodizing was carried out to the aluminum base material of the shape of a doughnut of this presentation on the same conditions as an example 1, and the porous anodic oxide film was formed. Homogenization of the barrier layer after coat formation was carried out.

[0036] Subsequently, the catalyst nucleus metal was deposited on the following conditions after rinsing by these conditions.

<Catalyst nucleus metal deposit conditions> (activation)

Liquid presentation SnCl₂ 4 g/LPdCl₂ 1 g/LHCl Whenever [10 mL/L solution temperature] 20 degree-C setup time 30 seconds (promotion-ized processing)

Liquid presentation NH₄ BF₄ Whenever [100 g/L solution temperature] 20 degree-C setup time Subsequently ultrasonic rinsing processing was carried out for 30 seconds, and flash plating was carried out on the following conditions after defecation.

<Flash plate plating condition> liquid presentation NiSO₄ and 6H₂O 26g / L hypophosphorous acid 30 g/LpH Whenever [4.5 solution-temperature] 60 degree-C time amount Subsequently it rinsed for 15 seconds and electroless deposition was carried out on the following conditions after defecation.

<Radio solution processing condition> liquid presentation nickel (sulfuric acid nickel) 3.0 g/L hypophosphorous acid 30 g/LCu (sulfuric acid Cu) 0.6 g/LpH Whenever [8.8 solution-temperature] 90 degree-C time amount Subsequently buffing was carried out for 120 minutes, 3 micrometers was removed, and surface roughness was finished.

[0037] Thus, the property same about the produced aluminum substrate as an example 1 was measured by the same approach. A result is shown in Table 1. Examples 1–3 have adhesion and shock resistance higher than the result of Table 1, and high display flatness is obtained by

surface slight polish, and the variation in anodic-oxidation-coatings thickness is small, and it turns out [heat-resistant] that it is.

[0038]

[Table 1]

表 1

	密着性	耐衝撃性	平坦度	陽極酸化皮膜の厚さ	膨れ発生
実施例 1	8	500 G	良好	20 μm	無
実施例 2	8	500 G	良好	20 μm	無
実施例 3	8	500 G	良好	20 μm	無
比較例 1	4	300 G	悪化	0~5 μm	有
比較例 2	4	300 G	悪化	0~5 μm	有
比較例 3	4	300 G	悪化	0~5 μm	有
比較例 4	10	200 G	良好	0 μm	無
比較例 5	8	500 G	良好	20 μm	有

[0039] (Example 1 of a comparison) Electroless deposition processing was performed and ground on the same conditions as an example 1, without having used the aluminum base material which deposited the catalyst nucleus metal obtained like the example 1, and giving flash plating. Thus, the property same about the created aluminum substrate as an example 1 was measured on these conditions. A result is shown in the same table 1 as an example.

(Example 2 of a comparison) Electroless deposition processing was performed and ground on the same conditions as an example 2, without having used the aluminum base material which deposited the catalyst nucleus metal obtained like the example 1, and giving flash plating. Thus, the property same about the created aluminum substrate as an example 1 was measured on these conditions. A result is shown in the same table 1 as an example.

(Example 3 of a comparison) Electroless deposition processing was performed and ground on the same conditions as an example 1, without having used the aluminum base material which deposited the catalyst nucleus metal obtained like the example 3, and giving flash plating. Thus, the property same about the created aluminum substrate as an example 1 was measured on these conditions. A result is shown in the same table 1 as an example.

(Example 4 of a comparison) Without using and carrying out anodizing of the aluminum base material of this presentation acquired like the example 1; the catalyst nucleus metal was deposited on an example 1 and these conditions, and, subsequently flash plating processing and non-electrolytic deposition processing were performed and ground. Thus, the property same about the created aluminum substrate as an example 1 was measured on these conditions. A result is shown in the same table 1 as an example.

[0040] the result of Table 1 to the example of a comparison (1-4) -- adhesion and shock resistance -- bad -- moreover -- the variation in anodic-oxidation-coatings thickness -- it is large, and there is the need of taking the large amount of polishes of a surface non-electrolyzed coat in obtaining good display flatness also with bad display flatness, and productivity is bad and it turns out that it becomes high [a manufacturing cost].

(Example 5 of a comparison) Except for using the electrolytic-solution presentation in anodizing conditions as a 5wt% sulfuric-acid water solution, the aluminum substrate was manufactured on the same conditions as an example 1, this substrate was heated on the same conditions as an example 1, and the existence of bulging generating was inspected visually. A result is shown in Table 1. The result of Table 1 shows that bulging occurs, although it is small.

[0041]

[Effect of the Invention] As mentioned above, the aluminum substrate concerning this invention The variation in anodic-oxidation-coatings thickness is small, and moreover are suitable for the

aluminum substrate for magnetic-recording media which can be used for the hard disk equipment of the portable type with which the load of the high impact is carried out since adhesion and shock resistance are high etc. The variation in anodic-oxidation-coatings thickness is small, and moreover the manufacture approach has adhesion and high shock resistance. It is the manufacture approach which is the aluminum substrate for magnetic-recording media which the amount of polishes of a surface non-electrolyzed coat could obtain display flatness good at least, was excellent in productivity, and was [be / which it is a manufacturing cost / it / low] excellent.

[Translation done.]

*** NOTICES ***

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.**** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

Drawing 1 Drawing 1 is the sectional view showing the structure of the substrate by this invention.

[Invention of a sign]

- 1 -- Aluminum base material
- 2 -- Anodic oxide film (porous membrane)
- 3 -- Metal used as a catalyst nucleus
- 4 -- Substrate metal flash plating film
- 5 -- P content nickel alloy electroless deposition film
- 6 -- Aluminum substrate
- 7 -- Hole (pore)

[Translation done.]

(19)日本国特許庁 (JP)

(12) 公開特許公報 (A)

(11)特許出願公開番号

特開2001-209925

(P2001-209925A)

(43)公開日 平成13年8月3日(2001.8.3)

(51) Int.Cl.⁷
 G 11 B 5/73
 C 23 C 18/32
 28/00
 C 25 D 11/04
 11/18

識別記号

F I
 G 11 B 5/73
 C 23 C 18/32
 28/00
 C 25 D 11/04
 11/18

テ-マコト⁸(参考)
 4 K 0 2 2
 4 K 0 4 4
 C 5 D 0 0 6
 Z 5 D 1 1 2
 A

審査請求 未請求 請求項の数11 OL (全9頁) 最終頁に続く

(21)出願番号 特願2000-17073(P2000-17073)

(71)出願人 000004743

日本軽金属株式会社
東京都品川区東品川二丁目2番20号

(22)出願日 平成12年1月26日(2000.1.26)

(72)発明者 小林 森
北海道苫小牧市晴海町43番地3 日本軽金属株式会社苫小牧製造所内(72)発明者 竹林 恒志
北海道苫小牧市晴海町43番地3 日本軽金属株式会社苫小牧製造所内(74)代理人 100077517
弁理士 石田 敏 (外3名)

最終頁に続く

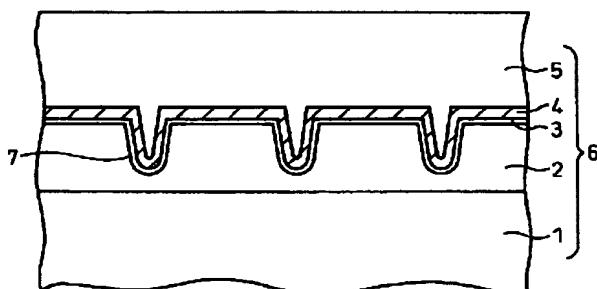
(54)【発明の名称】 磁気記録媒体用アルミニウム基板およびその製造方法

(57)【要約】

【課題】 耐衝撃性が高く、P含有Ni合金メッキ膜の基板に対する密着性が高い磁気記録媒体用アルミニウム基板を提供する。

【解決手段】 基材面にP含有Ni合金の無電解メッキ膜を備えたアルミニウム基板であって、アルミニウムからなる基材面に陽極酸化皮膜とその皮膜面に析出した触媒核金属とこの触媒核金属の析出した皮膜を実質的に覆う前記触媒核金属の組成と異なりかつ前記P含有Ni合金無電解メッキ膜の組成と異なる組成からなる下地金属フッ素めっき膜とこの下地金属めっき膜に無電解メッキした前記P含有Ni合金膜とを備えた磁気記録媒体用アルミニウム基板。

図1



【特許請求の範囲】

【請求項1】 アルミニウムからなる基材面にP含有Ni合金の無電解メッキ膜を備えたアルミニウム基板において、

アルミニウムからなる基材と、

該アルミニウム基材の陽極酸化により生成した多孔質皮膜と、

該多孔質皮膜面に析出した触媒核となる金属と、

該触媒核となる金属の析出した前記多孔質皮膜を実質的に覆う前記触媒核となる金属の組成と異なりかつ前記P含有Ni合金無電解メッキ膜と異なる組成の下地金属フラッシュめっき膜と、

該下地金属フラッシュめっき膜に無電解メッキした前記P含有Ni合金膜と、を備えたことを特徴とする磁気記録媒体用アルミニウム基板。

【請求項2】 前記陽極酸化により生成した多孔質皮膜の厚さが、0.5～50μmであることを特徴とする請求項1記載の磁気記録媒体用アルミニウム基板。

【請求項3】 前記多孔質皮膜が硫酸またはクロム酸電解液による陽極酸化皮膜であることを特徴とする請求項1または2に記載の磁気記録媒体用アルミニウム基板。

【請求項4】 前記多孔質皮膜面に析出した触媒核となる金属がPd, Cu, Ag, Sn, PまたはNiもしくはこれらの金属の中から選ばれた1又は2以上の金属を主成分とする合金であることを特徴とする請求項1から3までのいずれか1項に記載の磁気記録媒体用アルミニウム基板。

【請求項5】 前記下地金属フラッシュめっき膜はその厚さが5nm以上でしかもP含有量が3wt%以上10wt%未満のP含有Ni合金であることを特徴とする請求項2から4までのいずれか1項に記載の磁気記録媒体用アルミニウム基板。

【請求項6】 前記P含有Ni合金無電解メッキ膜はその厚さが0.5μm以上でしかもP含有量が10～13wt%であることを特徴とする請求項5に記載の磁気記録媒体用アルミニウム基板。

【請求項7】 アルミニウムからなる基材と、該アルミニウム基材の硫酸またはクロム酸電解液による陽極酸化により生成した厚さが0.5～50μmの多孔質皮膜と、

前記多孔質皮膜面に析出したPdと、

Pdの析出した前記多孔質皮膜を実質的に覆う厚さが5nm以上でP含有量が3wt%以上10wt%未満であるP含有Ni合金下地金属フラッシュめっき膜と、

該金属膜を覆う厚さが0.5μm以上でP含有量が10～13wt%であるP含有Ni合金膜と、を備えたことを特徴とする磁気記録媒体用アルミニウム基板。

【請求項8】 P含有Ni合金の無電解メッキ膜を備えたアルミニウム基板の製造方法において、

アルミニウムからなる基材に陽極酸化処理を施し多孔質

皮膜を形成する工程と、

該多孔質皮膜面に触媒核となる金属を析出させる工程と、

該触媒核となる金属の析出した前記多孔質皮膜を実質的に覆う前記触媒核となる金属の組成と異なりかつ前記P含有Ni合金無電解メッキ膜と異なる組成の下地金属フラッシュめっき膜を形成する工程と、

該下地金属フラッシュめっき膜を形成した前記多孔質皮膜に前記P含有Ni合金無電解メッキ膜を形成する工程と、

該P含有Ni合金無電解メッキ膜を研磨する各工程を経ることを特徴とする磁気記録媒体用アルミニウム基板の製造方法。

【請求項9】 前記陽極酸化処理を硫酸またはクロム酸電解液中で行うことを特徴とする請求項8に記載の磁気記録媒体用アルミニウム基板の製造方法。

【請求項10】 前記多孔質皮膜面に析出した触媒核となる金属がPd, Cu, Ag, Sn, PまたはNiもしくはこれらの金属の中から選ばれた1又は2以上の金属を主成分とする合金であることを特徴とする請求項8または9に記載の磁気記録媒体用アルミニウム基板の製造方法。

【請求項11】 アルミニウムからなる基材に硫酸またはクロム酸電解液中で陽極酸化処理を施す工程と、該陽極酸化により生成した多孔質皮膜面にPdを析出させる工程と、

該Pdの析出した前記多孔質皮膜に膜厚が5nm以上で燐含有量が3wt%以上10wt%未満であるP含有Ni合金下地金属フラッシュめっき膜を形成する工程と、

該下地金属フラッシュめっき膜の形成された前記多孔質皮膜を膜厚が0.5μm以上で燐含有量が10～13wt%であるP含有Ni合金無電解メッキ膜を形成する工程と、

該P含有Ni合金無電解メッキ膜を研磨する各工程を経ることを特徴とする磁気記録媒体用アルミニウム基板の製造方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】 本発明は、電子情報処理に用いられる固定型薄膜磁気記録ディスク（ハードディスク）等の高記録密度磁気記録媒体用のアルミニウム（本明細書において特に明示しない限りアルミニウムは、純アルミニウムおよびアルミニウム合金を含んでいるものである。）基材面上にP含有非晶質Ni合金膜を備えた磁気記録媒体用アルミニウム基板およびその基板の製造方法に関する。

【0002】

【従来の技術】 コンピュータなどの情報処理装置にはその外部記憶装置として“ハードディスクドライブ”もしくは“ハードディスク”と呼称される固定式の磁気記録

装置（磁気ディスク装置）が用いられている。一般に、この固定式磁気記録装置の記録媒体（磁気ディスク）の基板としては、アルミニウム基板が用いられている。この磁気記録媒体は通常、非磁性のアルミニウム基材を研削により平滑化し、表面清浄化および亜鉛置換処理の後無電解Ni-P合金メッキを行い、さらに研磨により平滑化し、テクスチャリング処理を行った後、非磁性金属下地層膜、磁性膜、保護膜、潤滑膜を順次形成して製造されている。

【0003】近年、携帯用コンピュータなどに搭載される可搬式の固定磁気ディスク装置に対応するため、作動時および移動時に磁気ヘッドとの衝突により傷付かないように、磁気ディスクに要求される耐衝撃性も年々高くなっている。それに伴い、上記のようにアルミニウム基材にP含有Ni合金メッキ膜を設けた基板では耐衝撃性が不十分になりつつある。そこで、アルミニウム基板に代わって、高価であり加工性は劣るが耐衝撃性に優れたガラス基板が使用され始めている。

【0004】無電解Niあるいは無電解P含有Ni合金メッキ膜を設けたアルミニウム基板には、多くの場合その表面に研磨により基板円周方向に沿って同心円状のテキスチャリングを施している。これは、記録再生用の磁気記録ヘッドと磁気記録媒体との間の吸着を防ぐと共に、磁気異方性を増加させるためである。さらに最近では、作動時のヘッド浮上量の著しい低減に伴い、基板のCSSゾーンにレーザービーム加工により微小突起を形成することも行われている。

【0005】しかし、ガラス基板の場合、硬くて加工性が悪いためテキスチャリングを施すことは困難である。しかも、一般的に透明であるためレーザービームの吸収性は悪く、また溶融温度も著しく高いため、特定形状の突起を均一に多数形成することも困難である。その対策として、テキスチャリングが可能であって、レーザービーム加工により微小突起を均一に形成できる耐衝撃性の高い基板として、ガラス面にPdを吸着させ、その上にP含有Ni合金メッキ膜を形成するガラス基板が提案（表面技術Vol.44 831~835 頁 1993）されているがガラスは加工性が悪く生産性に劣る。

【0006】アルミニウム基板は安価で加工性が良好で生産性が良いが、耐衝撃性が不十分である。特開昭62-3423号公報には、Al又はAl合金の陽極酸化皮膜の微細孔中に、金属を充填して触媒核とし、その上に強酸性液中でP含有Ni合金メッキ膜を形成した、P含有Ni合金メッキ膜の基板に密着性の良いアルミニウム基板が提案されている。

【0007】

【発明が解決しようとする課題】前記特開昭62-3423号公報に提案されているアルミニウム基板について検討したところ、微細孔中に金属を充填したアルミニウムの陽極酸化皮膜を強酸液中でP含有Ni合金メッキ膜

を形成する処理は、陽極酸化皮膜を著しく溶解し、皮膜表面の平滑性を低下させるばかりでなく皮膜厚さにむらを生じて、極端な場合には部分的に皮膜は完全に溶解、除去されてしまう。このため、研磨後の基板表面のうねり特性が得られなかったり、あるいは平滑なP含有Ni合金メッキ膜を得るにはメッキ膜の研磨量を大きくする必要がある。

【0008】また、磁気ディスク用下地めっき膜として用いられている10wt%以上のPを含有するNi-P合金用のめっき液は、析出初期の反応性が高いため、陽極酸化皮膜に直接析出させた場合良好な密着性が得られない。

【0009】

【課題を解決するための手段】即ち第1の発明は、アルミニウムからなる基材面にP含有Ni合金の無電解メッキ膜を備えたアルミニウム基板において、アルミニウムからなる基材と、該アルミニウム基材の陽極酸化により生成した多孔質皮膜と、該多孔質皮膜面に析出した触媒核となる金属と、該触媒核となる金属の析出した前記多孔質皮膜を実質的に覆う前記触媒核となる金属の組成と異なりかつ前記P含有Ni合金無電解メッキ膜と異なる組成の下地金属フラッシュめっき膜と、該下地金属フラッシュめっき膜に無電解メッキした前記P含有Ni合金膜とを備えたことを特徴とする磁気記録媒体用アルミニウム基板である。このような構造とすることによって、耐衝撃性が高く、P含有Ni合金メッキ膜の基板に対する密着性が高いアルミニウム基板が得られる。

【0010】アルミニウム基材の陽極酸化により生成した多孔質皮膜は、硬度の点では、P含有非晶質Ni合金膜とほぼ同程度かむしろ低い。しかし、弾性率は極めて高く弾性変形し難く、伸びはほとんどなく、降伏応力も大きいため塑性変形も生じ難いため、より大きな荷重に耐えられる。この多孔質皮膜の厚さを0.5μm以上とすることによって、耐衝撃性の高いアルミニウム基板が得られる。

【0011】またさらにこの陽極酸化皮膜が磷酸またはクロム酸電解液による多孔質皮膜であることによって、上記性質に加え、磁気記録ディスク製造過程で膨れのない耐熱性を持つアルミニウム基板が得られる。またさらにこの多孔質皮膜面に析出させる金属をPd、Cu、Ag、Sn、PまたはNiもしくはこれらの金属の中から選ばれた1又は2以上の金属を主成分とする合金とすることによって、P含有Ni合金メッキ膜の基板に対する密着性がより高いアルミニウム基板が得られる。

【0012】またさらに下地金属フラッシュめっき膜をその厚さが5nm以上でしかもNi合金メッキ膜にすることにより基板に対する密着性がより高いアルミニウム基板が得られる。またさらにP含有Ni合金無電解メッキ膜はその厚さが0.5μm以上でしかもP含有量が10~13wt%とすることによって、硬度の高い非磁性のア

ルミニウム基板が得られる。

【0013】第2の発明は、P含有Ni合金の無電解メッキ膜を備えたアルミニウム基板の製造方法において、アルミニウムからなる基材に陽極酸化処理を施し多孔質皮膜を形成する工程と、該多孔質皮膜面に触媒核となる金属を析出させる工程と、触媒核となる金属の析出した前記多孔質皮膜に前記触媒核となる金属の組成と異なるかつ前記P含有Ni合金無電解メッキ膜と異なる組成の下地金属フラッシュめっき膜を形成する工程と、該下地金属フラッシュめっき膜を形成した前記多孔質皮膜に前記P含有Ni合金無電解メッキ膜を形成する工程と、該P含有Ni合金膜を研磨する各工程を経ることを特徴とする磁気記録媒体用アルミニウム基板の製造方法である。このような製造方法とすることによって、多孔質皮膜面の溶解され難く、P含有Ni合金無電解メッキ膜の研磨量を少なくして生産性良く、耐衝撃性の高く、P含有Ni合金無電解メッキ膜の基板に対する密着性が高いアルミニウム基板を製造することができる。

【0014】この多孔質皮膜の厚さを20μm以上とすることにより、さらに耐衝撃性の高いアルミニウム基板を製造することができる。この陽極酸化処理を塩酸またはクロム酸電解液中で行うことにより、上記効果に加え磁気記録ディスク製造過程で膨れのないアルミニウム基板を製造することができる。

【0015】この多孔質皮膜面にPd, Cu, Sn, AgまたはNiもしくはこれらの金属の中から選ばれた1又は2以上の金属を主成分とする合金を析出させることによって、上記効果に加えP含有Ni合金メッキ膜の基板に対する密着性がより高いアルミニウム基板を製造することができる。この下地金属フラッシュめっき膜を形成する工程のメッキ液は好ましくは弱アルカリ～弱酸性であり、膜厚が5nm以上とすることにより、上記効果に加え多孔質皮膜面の溶解され難く、P含有Ni合金無電解メッキ膜の研磨量を少なくして生産性良く、耐衝撃性高く、P含有Ni合金無電解メッキ膜の基板に対する密着性が高いアルミニウム基板を製造することができる。

【0016】このP含有Ni合金無電解メッキ膜を形成する工程のメッキ液が酸性であり、膜厚が0.5μm以上で含有量が1.0～1.3wt%である非晶質であることにより、上記効果に加え多孔質皮膜面の溶解され難く、硬度の高いアルミニウム基板を製造することができる。

【0017】

【発明の実施の形態】図1は、本発明の磁気記録媒体用のアルミニウム基板の一実施形態を示すもので、ここに示すアルミニウム基板6は、アルミニウム基材1上に該アルミニウムを陽極酸化して形成した多孔質皮膜2、該皮膜2上に析出させた触媒核となる金属3、該触媒核となる金属3の析出した多孔質皮膜2を覆う下地金属フラッシュめっき膜4、該下地金属フラッシュめっき膜4を覆うP含有Ni合金無電解メッキ膜5が順次形成された

ものである。ここで下地金属メッキ膜4は触媒核となる金属3と異なりかつP含有Ni合金メッキ膜5と組成の異なる金属である。7は陽極酸化皮膜2中の孔（ポア）である。

【0018】アルミニウム基材としては、その組成を限定しないが、Alの他一般的なAl-2～5wt%Mg系合金が固溶強化タイプで、晶出物少なく機械的な強度付与ができる。多孔質陽極酸化皮膜2は、酸性液中でアルミニウム基材を陽極として直流陽極酸化法によって形成される。該陽極酸化皮膜2は硬質で耐衝撃性が高く、アルミニウム基材の軟質性をカバーして可搬性磁気記録媒体のような場合に好ましくなる。特にこの厚さを0.5μm以上とすると、アルミニウム基材の軟質さをカバーリ耐衝撃性を付与する。好ましくは20μmを超えることが好ましい。

【0019】磁気記録ディスクは、アルミニウム基板をテキスチャ加工後Cr膜をスパッタ法で設け、更に同法でCo等の磁気記録媒体を設けるが、この場合アルミニウム基板が250～300°Cに昇温するので、この多孔質皮膜が塩酸またはクロム酸陽極酸化皮膜であると、該皮膜中の結晶水および付着水量が少なく、前記加熱に耐え、耐熱性を持たせることができて好ましい。

【0020】陽極酸化皮膜2の表面粗さは特に限定されないが、過度に大きい場合は、P含有Ni合金無電解メッキ膜の厚さを厚くメッキしてアルミニウム基板面の研磨をすることになり、研磨に時間がかかり、コストと生産性が悪くなるので、表面粗さはRaで500Å以下としておくと好ましい。多孔質陽極酸化皮膜2の上に析出させた触媒核金属3は、その上にメッキした下地金属フラッシュめっき膜4と強固に結合し、剥離し難くなつて耐久性に優れる。この触媒核金属3は、特にPd, Ag, Sn, P, CuまたはNiもしくはこれらのの中から選ばれた1又は2種以上を主成分とする合金であると多孔質陽極酸化皮膜2面に比較的に均一に析出し下地金属フラッシュめっき膜4と強固に結合する。ここで主成分とはこれらの金属が50wt%以上含有していることをいう。この強固になるのは、多孔質陽極酸化皮膜の表面および孔内に下地金属フラッシュめっき膜が析出することによるアンカー効果によるものと考えられる。特にPdであると触媒性が高く、孔内の析出も十分に行われ好ましい。

【0021】十分な耐剥離性を持たせるには下地金属フラッシュめっき膜4の厚さは5nm以上のNi合金で、P含有量を3wt%以上10wt%未満とし韌性を付与し、P含有Ni合金無電解メッキ膜5をその厚さは0.5μm以上で、P含有量を1.0～1.3wt%とすることにより、強固に結合し、硬度を有し、非磁性、を持つことになる。しかもP含有Ni合金無電解メッキ膜5の膜厚さを0.5μm以上とすることで、テキスチャ加工することができる。

【0022】このような構成としたアルミニウム基板は、密着性高く耐剥離性、耐衝撃性が優れる。この後アルミニウム基板は、テキスチャーリング加工し、非磁性下地膜としてCr, Ti, Ni, Si, Ta, W、またはこれらの中少なくとも1種を主成分とする合金とする膜を設ける。

【0023】この非磁性下地膜の上に、CoまたはCoを主成分とする合金をスパッタ法等の方法で磁性膜を設ける。この磁性膜の上に、保護膜としてカーボン等の膜を設けアルミニウム製磁気記録再生用基板とされる。次に製造方法について説明する。

【0024】純Alや2~5wt%Mgを含有するAl-Mg系等のアルミニウム板をプレスで打ち抜いてドーナツ状のアルミニウム板を作成し、プレス癖を除去するために、打ち抜かれたドーナツ状アルミニウム板を積層加圧し、250~350°Cに加熱する。爾後平坦さを出すために研削する。研削後、脱脂、酸洗浄、水洗浄、等の前処理後、硫酸、磷酸、硝酸、クロム酸および酒石酸の無機、有機の酸からなる群から選択した1種または2種以上の酸を含む好ましくはpH3以下の強酸性水溶液中で、アルミニウム基板を陽極として直流陽極酸化処理を施すと多孔質型陽極酸化皮膜が得られる。酸の濃度は酸種によって異なるが概ね0.5~35%である。温度は好ましくは5~30°Cで低温とすると皮膜硬度が増す。皮膜厚さは印可電気量で制御できる。酸の種類、電解条件は皮膜の耐衝撃性に大きな影響はない。陽極酸化処理後、特に必要はないが、陽極酸化処理皮膜の底部に在る無孔質酸化皮膜、所謂バリア層の厚さを均一化してもよい。そのために、たとえば、陽極酸化処理の同じ電解液中で陽極酸化処理終了後印可電圧を下げる方法等を施してもよい。あるいは、陽極酸化処理後中性溶液中、望ましくはpH4~9の中性塩電解液中で、定電流電解によって50Vまで昇圧し、アルミニウム基材と多孔質陽極酸化皮膜との間に、厚さが約70nmの無孔質陽極酸化を設けてもよい。また、二次電解析出による孔内への金属析出処理を施してもよい。

【0025】磷酸またはクロム酸電解による処理は、得られた陽極酸化皮膜中の含水率を低くすることができて好ましい。また得られた皮膜は耐熱性があり、磁気記録ディスク製造過程で膨れのない耐熱性を持つアルミニウム基板が得られる。陽極酸化処理後触媒核となる金属を析出させる。触媒核となる金属を析出させる前に、該陽極酸化処理基材を硝酸、磷酸等の酸もしくは苛性ソーダ等のアルカリで洗浄してもよく、平砥石、ブラシ、ショットブラスト、バフ等で機械的研磨をしてもよく、硝酸、磷酸等の酸でエッチングして研磨してもよく、また、化学的方法でエッチングして粗面化あるいは機械的方法で粗面化し、または両方法を採用して表面粗さを調整する。いずれにしろ多孔質陽極酸化皮膜面の平均粗さはRaで500Å以下、好ましくは100Å以下、さ

らに好ましくは50Å以下としておくとP含有Ni合金無電解メッキ膜との密着性を向上させかつ研磨量を少なくできてよい。研削研磨した場合はその後に研削研磨滓を除去洗浄する。アルミニウム基材研削後からP含有Ni合金無電解メッキ処理後の仕上げ研磨の間に歪み取り、脱水、脱ガスあるいは結晶化のために100°C~300°Cの熱処理を行っても良い。

【0026】陽極酸化皮膜への触媒核金属の析出は、電解法あるいは不導体上への触媒付与法等が採用できる。例えば陽極酸化処理したアルミニウム基材を触媒核金属塩の水溶液中に浸漬して金属電解、無電解あるいは吸着により多孔質皮膜面に析出させる。この場合触媒核金属の析出の前処理として、還元作用のある金属の水溶液中に陽極酸化処理したアルミニウム基材を浸漬して還元性金属を一旦多孔質皮膜面に付着させておき、この還元力で爾後の前記金属塩の水溶液中に浸漬して触媒核金属を多孔質皮膜面で還元し触媒核金属としたり、あるいは前記の前処理を施すことなくアルミニウム基材を触媒核金属塩の水溶液中に浸漬して金属塩を多孔質皮膜面に付着させ、加熱や他の手段で触媒核金属とする方法等が採用できる。

【0027】析出させる触媒核となる金属は特に限定されないが、特にPd, Cu, Ag, Sn, PまたはNiもしくはこれらの中から選ばれた1又は2種以上を主成分とする合金であって、上述の如くこれらの塩化物、硝酸塩等の水溶液中に前記研削洗浄後の陽極酸化処理したアルミニウム基材を浸漬して該触媒核金属を多孔質皮膜面に析出させる。

【0028】触媒核金属が析出したアルミニウム基材を洗浄した後、メッキするフラッシュメッキ膜の形成は、金属塩を高濃度に溶解させたアルカリもしくは酸水溶液中で、比較的短時間で例えば5~180秒程度の時間でメッキする形成方法で、このような方法で触媒核となる金属の組成と異なりかつ表面のP含有Ni合金無電解メッキ膜と異なる組成の金属を、触媒核金属が析出した多孔質皮膜全面を覆うことによって、次工程の無電解メッキ処理液で多孔質皮膜を溶解させることなく、また析出させた触媒核金属でメッキ液を汚すことがなく純粋な状態で無電解メッキ処理をすることができる。フラッシュメッキのメッキ液がNi塩とP塩を含むメッキ液であるとP含有Ni合金無電解メッキ液を汚染せずに処理できる。フラッシュメッキの液温度は比較的低温度、例えば20~50°Cでメッキすると下地材の溶解を抑えることができ、メッキ液の汚染を低めることができる。このフラッシュメッキ液の液性を弱アルカリ乃至弱酸性液、例えばpH表示で4.0~10.0とすると多孔質型陽極酸化皮膜の溶解量が少なく、皮膜が平滑さ、酸化皮膜自体を失うことがなく好ましい。P含有Ni合金をフラッシュメッキする場合は、メッキ浴としては、例えば、硫酸ニッケル、塩化ニッケル、等のニッケル源と、次亜硝酸

塩等の焼源を含むものを採用することができる。

【0029】 フラッシュメッキ膜を形成した基材は次にP含有Ni合金膜を無電解メッキ法で形成する。この無電解メッキでP含有の非晶質Ni合金膜が得られる。ここで、無電解メッキされたP含有非晶質Ni合金膜とはNi, P以外に第3の元素例えばCuを含んだものも含むものである。該P含有非晶質Ni合金膜を形成するには、液性を強酸性とし焼源濃度を高めてP含有非晶質Ni合金膜を形成する。Ni源とP源はメッキ液としては、例えば、硫酸ニッケル、塩化ニッケル、等のNi源と、次亜塩酸塩等のP源を含むものを採用することができる。濃度はNi源が4g/L~8g/L、P源が次亜リン酸として20g/L~40g/L、温度は60°C~100°Cから適宜選択する。

【0030】 P含有非晶質Ni合金膜の厚さを0.5μm以上にするには処理時間を3分以上とし、P含有量を1.0~1.3wt%にするにはメッキ液組成、析出速度を管理することにより行うことができる。

【0031】

【実施例】(実施例1) Al-4.5wt%Mg合金板をプレス機で打ち抜いたドーナツ状のアルミニウム基材を使用し、歪焼純して平坦度を出した後、アルカリ液で脱脂し、硫酸酸性液で表面の酸化物を除去し、水洗して清浄化した。次いで下記条件で陽極酸化処理して多孔質皮膜を形成した。皮膜形成後バリアー層の均質化処理をした。

<陽極酸化処理条件>

電解液組成	5wt%草酸水溶液
直流定電流電解	1.2A/dm ²
皮膜厚さ	20μm
液温度	30°C設定

次いで純水で3回水洗後下記条件で触媒核金属を析出させた。

<触媒核金属析出条件>

液組成	
PdCl ₂	5g/L
HCl	10mL/L
液温度	20°C設定
時間	30秒

次いで超音波水洗処理して清浄化後下記条件でフラッシュメッキした。

<フラッシュメッキ処理条件>

液組成	
NiSO ₄ ·6H ₂ O	25g/L
次亜塩酸	25g/L
pH	7
液温度	40°C
時間	15秒

次いで水洗して清浄化後下記条件で無電解メッキした。

<無電解処理条件>

液組成

NiSO ₄ ·6H ₂ O	26g/L
次亜塩酸	30g/L
pH	4.5
液温度	90°C
時間	120分

次いでバフ研磨し3μm除去して表面粗さを仕上げた。

【0032】このようにして作製したアルミニウム基板について下記方法で密着性、耐衝撃性、平坦度、陽極酸化皮膜の厚さのバラツキを測定した。また270°Cに10秒間加熱し目視観察で膨れ発生の有無を検査した。結果を表1に示す。

<密着性>

JIS K 5400(1990) 塗料一般試験方法
すきま間隔5mm、ます目の数9となるようにカッターナイフでアルミニウム地に達するまで切り込みを入れ、その上にテープを貼りつけ、このテープを急激に剥がし、表面の剥がれた状態を基盤目試験の評価点数0~10で表示（評価点数が高いほど密着性の良いことを表す）した。

<耐衝撃性>吉田精機社製 振子式衝撃試験機（型式PS-T-300）を用いて基板面に衝撃を与え、凹みの有無を目視判定した。

<平坦度>溝尻光学工業所（株）製 干渉縞平坦度検査器（型式FT-100）を用いて基板の水平置き平坦度を測定した。

<陽極酸化皮膜の厚さのバラツキ>JIS H 8680-2(1998) 陽極酸化皮膜厚さ試験方法フィッシャーインスツルメンツ（株）製 膜厚測定器（型式フィッシャースコープMMS）を用いて陽極酸化皮膜の厚さについて測定を行った。

【0033】結果を表1に示す。

(実施例2) 実施例1と同じ条件で同組成のドーナツ状のアルミニウム基材に陽極酸化処理して多孔質皮膜を形成した。皮膜形成後バリアー層の均質化処理をした。次いで同条件で水洗後下記条件で触媒核金属を析出させた。

<触媒核金属析出条件>

(感受性化処理)

液組成	
SnCl ₂	10g/L
HCl	10mL/L
液温度	20°C設定
時間	30秒

(活性化処理)

液組成	
PdCl ₂	5g/L
HCl	10mL/L
液温度	20°C設定
時間	30秒

次いで超音波水洗処理して清浄化後実施例1と同じ条件でフラッシュメッキした。

【0034】次いで水洗して清浄化後下記条件で無電解メッキした。

<無電解メッキ処理条件>

液組成

Ni (硫酸Ni) 3.0 g/L

次亜塩酸 30 g/L

Cu (硫酸Cu) 0.6 g/L

pH 8.8

液温度 90°C

時間 120分

次いでバフ研磨し3μm除去して表面粗さを仕上げた。

【0035】このようにして作製したアルミニウム基板について実施例1と同じ特性を同じ方法で測定した。結果を表1に示す。

(実施例3) 実施例1と同じ条件で同組成のドーナツ状のアルミニウム基材に陽極酸化処理して多孔質皮膜を形成した。皮膜形成後バリアー層の均質化処理をした。

【0036】次いで同条件で水洗後下記条件で触媒核金属を析出させた。

<触媒核金属析出条件>

(活性化処理)

液組成

SnCl₂ 4 g/L

PdCl₂ 1 g/L

HCl 10mL/L

液温度 20°C設定

時間 30秒

(促進化処理)

液組成

NH₄BF₄ 100 g/L

液温度 20°C設定

時間 30秒

次いで超音波水洗処理して清浄化後下記条件でフラッシュメッキした。

<フラッシュめっき条件>

液組成

NiSO₄ · 6H₂O 26 g/L

次亜塩酸 30 g/L

pH 4.5

液温度 60°C

時間 15秒

次いで水洗して清浄化後下記条件で無電解メッキした。

<無電解処理条件>

液組成

Ni (硫酸Ni) 3.0 g/L

次亜塩酸 30 g/L

Cu (硫酸Cu) 0.6 g/L

pH 8.8

液温度 90°C

時間 120分

次いでバフ研磨し3μm除去して表面粗さを仕上げた。

【0037】このようにして作製したアルミニウム基板について実施例1と同じ特性を同じ方法で測定した。結果を表1に示す。表1の結果より、実施例1~3は密着性および耐衝撃性が高く、表面の僅かな研磨で高い平坦度が得られ、陽極酸化皮膜厚さのバラツキが小さく、また耐熱性のあることが判る。

【0038】

【表1】

表1

	密着性	耐衝撃性	平坦度	陽極酸化皮膜の厚さ	膨れ発生
実施例1	8	500G	良好	20 μm	無
実施例2	8	500G	良好	20 μm	無
実施例3	8	500G	良好	20 μm	無
比較例1	4	300G	悪化	0~5 μm	有
比較例2	4	300G	悪化	0~5 μm	有
比較例3	4	300G	悪化	0~5 μm	有
比較例4	10	200G	良好	0 μm	無
比較例5	8	500G	良好	20 μm	有

【0039】(比較例1) 実施例1と同様にして得られた触媒核金属を析出させたアルミニウム基材を使用し、フラッシュメッキを施すことなく、実施例1と同じ条件で無電解メッキ処理を施し研磨した。このようにして作

成されたアルミニウム基板について実施例1と同じ特性を同条件で測定した。結果を実施例と同じ表1に示す。

(比較例2) 実施例1と同様にして得られた触媒核金属を析出させたアルミニウム基材を使用し、フラッシュメ

ツキを施すことなく、実施例2と同じ条件で無電解メッキ処理を施し研磨した。このようにして作成されたアルミニウム基板について実施例1と同じ特性を同条件で測定した。結果を実施例と同じ表1に示す。

(比較例3) 実施例3と同様にして得られた触媒核金属を析出させたアルミニウム基材を使用し、フラッシュメッキを施すことなく、実施例1と同じ条件で無電解メッキ処理を施し研磨した。このようにして作成されたアルミニウム基板について実施例1と同じ特性を同条件で測定した。結果を実施例と同じ表1に示す。

(比較例4) 実施例1と同様にして得られた同組成のアルミニウム基材を使用し、陽極酸化処理することなく、実施例1と同条件で触媒核金属を析出させ、次いでフラッシュメッキ処理、無電解析出処理を施し研磨した。このようにして作成されたアルミニウム基板について実施例1と同じ特性を同条件で測定した。結果を実施例と同じ表1に示す。

【0040】表1の結果から比較例(1~4)は密着性および耐衝撃性悪く、しかも陽極酸化皮膜厚さのバラツキ大きく、平坦度も悪く良好な平坦度を得るには表面の無電解皮膜の研磨量を大きく取る必要が有り生産性悪く、製造コストの高くなることが判る。

(比較例5) 陽極酸化処理条件における電解液組成を5wt%硫酸水溶液とすることを除いて実施例1と同じ条件でアルミニウム基板を製作し、この基板を実施例1と同

じ条件で加熱し膨れ発生の有無を目視検査した。結果を表1に示す。表1の結果から僅かではあるが膨れが発生することが判る。

【0041】

【発明の効果】 上述した如く、本発明に係るアルミニウム基板は、陽極酸化皮膜厚さのバラツキが小さくしかも密着性および耐衝撃性が高いから高い衝撃の負荷される可搬式の固定磁気ディスク装置等に使用できる磁気記録媒体用のアルミニウム基板に適し、またその製造方法は、陽極酸化皮膜厚さのバラツキが小さくしかも密着性および耐衝撃性が高く、表面の無電解皮膜の研磨量が少なくとも良好な平坦度を得ることができ、生産性が優れ製造コストの低い等の優れた磁気記録媒体用のアルミニウム基板の製造方法である。

【図面の簡単な説明】

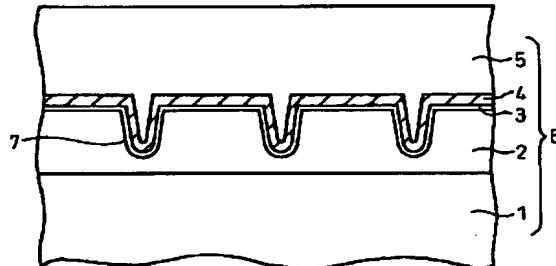
【図1】 図1は、本発明による基板の構造を示す断面図である。

【符号の発明】

- 1…アルミニウム基材
- 2…陽極酸化皮膜(多孔質膜)
- 3…触媒核となる金属
- 4…下地金属フラッシュメッキ膜
- 5…P含有Ni合金無電解メッキ膜
- 6…アルミニウム基板
- 7…孔(ポア)

【図1】

図1



フロントページの続き

(51) Int.Cl. 7
G 11 B 5/84

識別記号

F I
G 11 B 5/84

マーク(参考)

Z

(72) 発明者 島田 英樹
北海道苫小牧市晴海町43番地3 日本軽金
属株式会社苫小牧製造所内

(72) 発明者 高橋 武良
北海道苫小牧市晴海町43番地3 日本軽金
属株式会社苫小牧製造所内

F ターム(参考) 4K022 AA02 AA31 AA44 BA14 BA16
CA06 CA17 CA18 CA19 CA20
CA21 CA28 DA01
4K044 AA06 BA06 BA12 BA15 BB03
BB05 BB13 BC05 CA15 CA17
5D006 CA01 CA05 CA06 CB04 CB07
CB08 FA07
5D112 AA02 AA24 BA06 EE01 GA29